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TITLE:	New Techniques in Nondestructive Testing by Acoustical and Exo-electron Emission

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ACOUSTICAL EMISSION STUDIES

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Deforming materials emit high-frequency acoustical waves. The mechanism or mechanisms responsible for the initiation of these waves remains relatively unknown. Evidence has been produced to indicate that the waves originate in the bulk of a body and are probably connected with the formation of microcracks at stress concentrations and other crack nucleation sites.

One of the most important problems connected with making use of acoustical emission data is the determination of the significance of the various sounds emitted. Studies so far have concentrated on recording the number of transient emissions occurring in a certain frequency range per unit time and correlating this information with the mechanical state of the deforming specimen. The most popular frequency range seems to lie below 5 MHz.

The purpose of the present investigation is to determine two points: (1) the upper range of the frequency spectrum of acoustical emissions and whether it is limited by the rapidly rising attenuation usually associated with rising frequency and (2) the existence of any recognizible peaks or valleys in the acoustical power spectrum identifiable with a particular mechanism of acoustical emission, for example, with initial microcrack size.

A sample calculation shows that high-frequency stress waves should be expected from microcrack formation and propagation. Depending upon the nature of the stress concentration, frequencies as high as 100 MHz should be expected. Furthermore, as cracks extend, there should be a correspon-ding decrease in the frequencies of the maximum in the power spectrum.

In the present study, the acoustical output of quartz crystal trans-ducers attached to deforming specimens has been analyzed by a Hewlett Packard spectrum analyzer. This method does not permit the frequency con-tent of individual transient pulses (emission) to be measured. It can, however, measure the average frequency spectrum of several hundred pulses. At present, this experimental constraint seriously restricts the type of specimens that can be studied. More recent work, at present incomplete, has centered on a method of measuring the frequency spectrum of individual pulses.

SPECTRUM ANALYZER STUDIES

The Hewlett Packard spectrum analyzer is essentially a frequency swept, very high gain receiver. It has provisions for varying both the sweep frequency range and the local frequency of sweeping. Given the sweep range, only a limited number of sweeping rates are possible which will allow calibrated operation. Thus if a transient lasting only one milli-second is introduced into the receiver while sweeping at one millisecond per 1 MHz frequency range, then depending on the frequency content of the pulse, the analyzer may record power received in the 1 MHz frequency

window or not. Only if a number of transients is received and superimposed can a representative spectrum be constructed. The Hewlett Packard analyzer being used has a variable persistent screen which enables such a superposition. Figure 1 is a typical frequency spectra of transient pulses from a model of microcrack propagation.

Figure 1 shows the spectrum of acoustical emission from the separation of adhesive layers. The source of the emission is the ductile fracturing of many highly deformed elastic bands of the order of 10^{-3} cm in cross-section. As can be seen, most of the acoustical power lies below 3 MHz with less than 1 per cent above 5 MHz. The crystal cutoff is at 10 MHz. Increase in the rate of "macro-crack" growth is controlled by increasing the tensile stress tending to cause separation of the layers. Notice the expected shift in the frequencies to higher values with increased macro-crack velocity. Figure 2 shows the block diagram for the measurements made in Figure 1.

FUTURE WORK

The development of a single-shot detector using an array of quartz crystals operating with high Q's is continuing. The first model will use only a three-element array, with the highest crystal fundamental being at 30 MHz. Bend tests on precracked, high yield-stress steel will be performed in an attempt to confirm the prediction that the first stage of fracture is preceded by high-frequency components of acoustical emission.

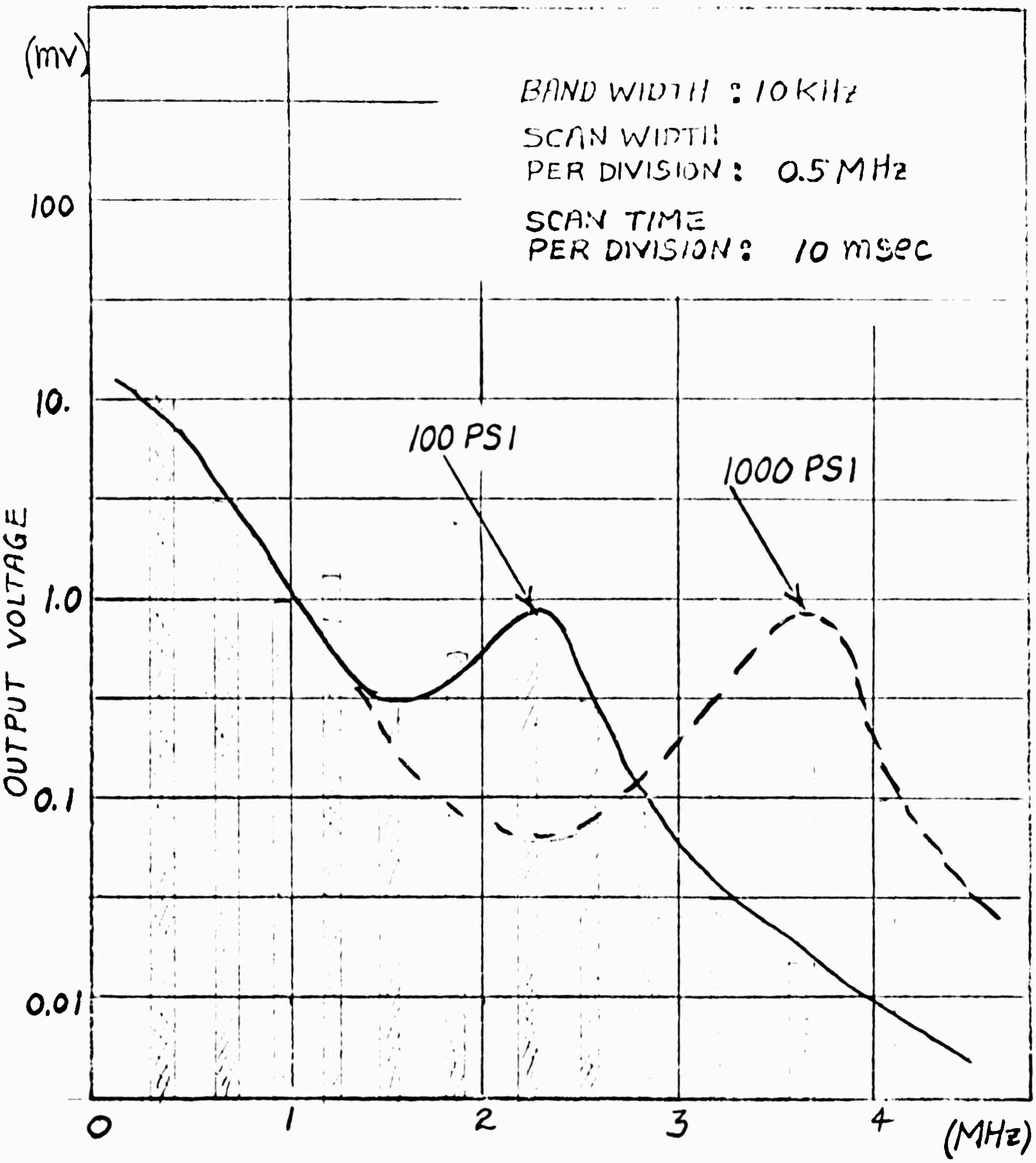

FIGURE 1. ACOUSTICAL EMISSION SPECTRUM OF ADHESIVE LAYER SEPARATION MODEL OF CRACK PROPAGATION

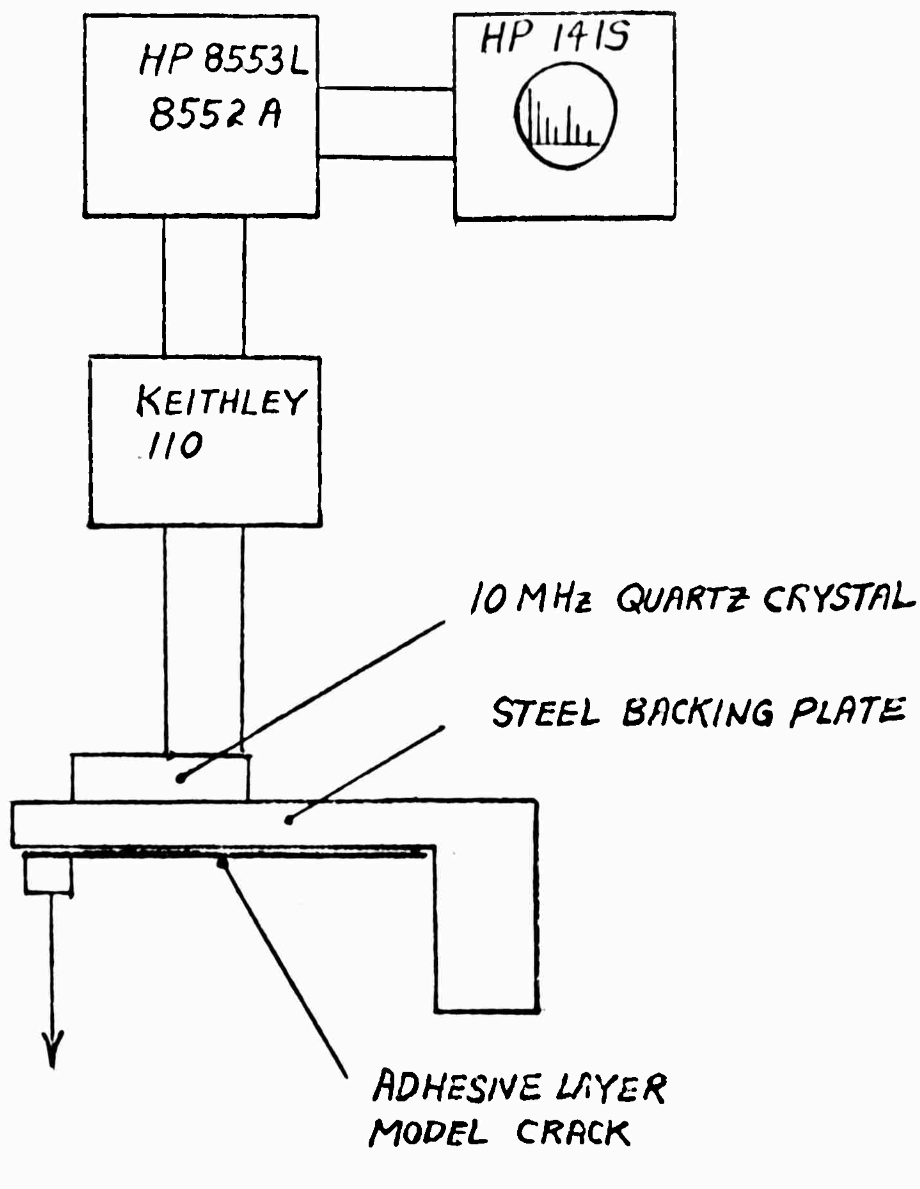

FIGURE 2. BLOCK DIAGRAM OF SPECTRUM ANALYZER SYSTEM

EXO-ELECTRON EMISSION FROM METALS UNDER STRESS

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INTRODUCTION

In the original proposal it was suggested that a relation existed between dislocations and exo-electron emission. If true, this would indicate that before gross failure occurred, the multiplication and movement of dislocations would provide a pre-failure current of exo-electrons which might serve as pre-fracture warning.

The literature of exo-electron emission indicated that the phenomenon was not clearly understood. Various experimenters (1, 2) had related the emitted electrons to such things as oxide layers, adsorption of gases, dislocations, and ambient light intensity. It was clear that a primary step would be a determination of the exact relation, if any, between exo-electron emission and dislocations.

The apparatus built for this experiment is an improved and redesigned version of the field emission system used in our earlier work. The system is shown in Figure 1. Vacuums of 10^{-9} torr are obtained with a combination of mechanical, oil, and Vac-Ion pumping. Sorption pumps are available

if the experimental system must be isolated from the mechanical pumps

The applied stress is measured with an Instron Load Cell.* The applied electrostatic fields are shaped by a removable inner grid and a conductive phosphor screen. The screen allows us to operate the system in the field emission mode if we wish to examine a specimen for micro-crack formation before gross failure occurs. Gas handling valves, pressure gauges, and bake-out heaters are available to admit controlled amounts of gas and permit rapid attainment of ultra-high vacuums.

Electronic apparatus for measurement and recording of strain gauge output and electron current is available. Careful design of the electrical system by Mr. W. Ott and the use of operational amplifiers** allow us to measure electron currents of less than 10^{-12} amps.

EXPERIMENTS AND RESULTS

The specimens to date have been type-A nickel wire nominally 0.032" in diameter by 3" long and etched in 40 per cent HCl solution to about 0.020" diameter to insure that fracture will occur away from the grips. In a typical experiment the wire is pulled to failure at a rate of 0.025"

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*Donated by the Instron Engineering Company of Canton, Massachusetts, through the courtesy of Mr. A. Cozens.

**The cooperation of Mr. Thomas Fern and the Burr-Brown Research Corporation of Tucson, Arizona, in providing operational amplifiers and applications information is greatly appreciated.

per second. The stress and electron current are recorded from the time the initial stress is applied until failure occurs.

Typical results are shown in Figure 2 where we have plotted stress and electron current to the same time scale.* There is a distinct time correlation between stress and electron current. We are not prepared at this time to present an explanation of the two phases observed in the exo-electron current. The early constant-current period we have called Phase I; the rapid rise, we call Phase II. Phase I may be connected with the elastic part of the stress cycle; Phase II may be evidence of plasticity at fracture, but this is mere speculation at the moment.

Other experimental studies have been devoted to the effect, if any, of ambient oxygen on exo-electron emission. For these studies the nickel specimens were heated to 1000^{o}C in a vacuum of 5×10^{-7} torr for 5 hours. The wires were cooled in the vacuum and then the system was flushed 10 times with hydrogen by filling it to one atmosphere and pumping it down to 5×10^{-7} torr. The actual tensile test was run at a pressure of 5×10^{-8} torr; we estimate that the partial pressure of oxygen was about 10^{-10} torr.

Other tensile test experiments were run at varying oxygen pressures for comparison. The results indicate that there is no apparent relation between the exo-electron current and the ambient oxygen pressure. This is

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*The data of Figure 2 is a composite of the results of two different experiments because the necessary apparatus for simultaneous recording of stress and electron current was under construction at the time.

in agreement with the results of reference (2). There may be an effect of residual oxides on the specimen surface because we cannot be sure that the heating - hydrogen cycle removed all oxides from the nickel. In any case the evidence of a lack of effect of ambient oxygen is significant and will be investigated with other metals.

FUTURE PLANS

We plan to continue improving the apparatus with the objective of studying the details of the exo-electron current and its relation to elastic and plastic strain. We will stop the pulling system during Phase I and examine the specimen for evidence of ductile strain and micro-cracks. The effect of oxide layers on exo-electron emission will be explored with oxide-coated specimens and with specimens which have been stripped of all oxide layers.

REFERENCES

(1) H. J. Mueller, "Exo-Electron Emission and Related Electron Emissions," AD 276, 213, Dec. 1969.
(This is an excellent review of the work through 1961.)

(2) J. A. Ramsey, "The Emission of Electrons from Aluminum Abraded in Atmospheres of Air, Oxygen, Nitrogen, and Water Vapor," *SURFACE SCIENCE* 8, No. 3, 313-323, 1967.

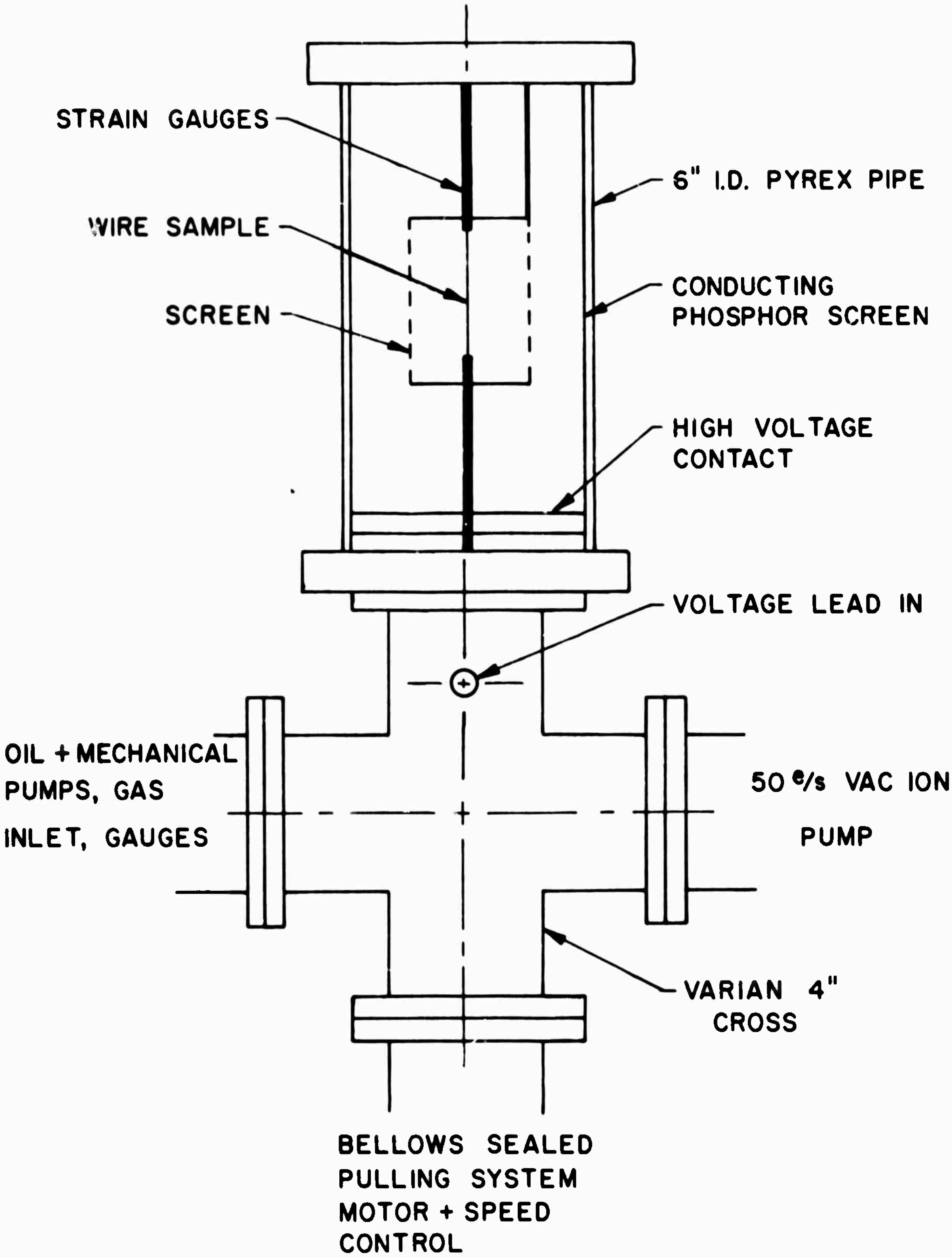

Figure 1. Exo-Electron Experimental System

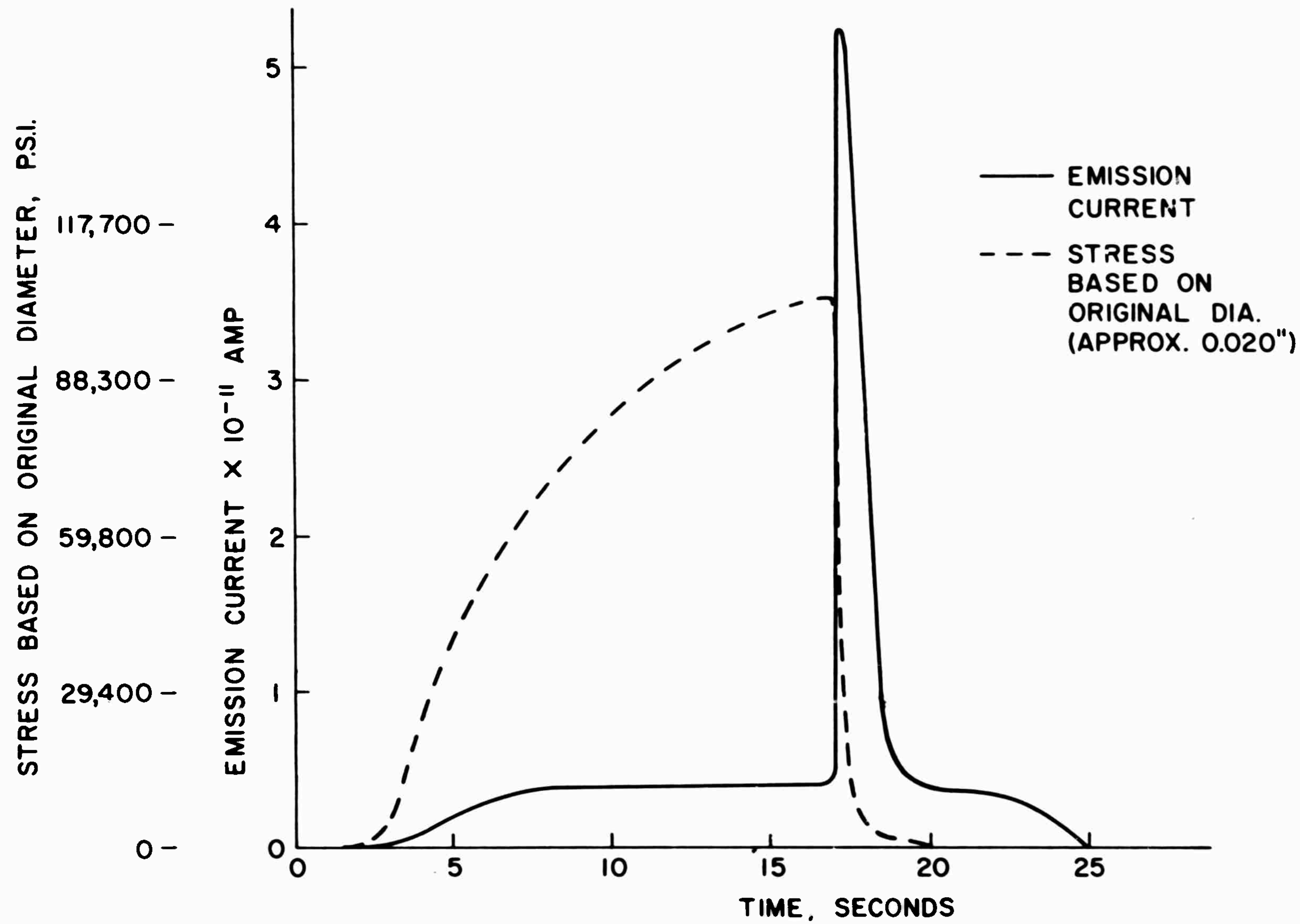

Figure 2. Applied Stress and Exo-Electron Current *vs.* Time